Electronic Version

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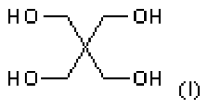
Claims

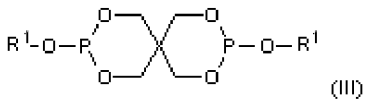
[c1] 1. An improved method for synthesizing pentaerythritol diphosphites comprising the steps of:

(a) transesterifying pentaerythritol of formula (I)



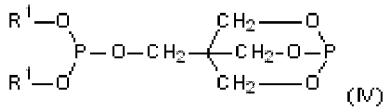
(I)

with a monophosphite to form a first reaction mixture which comprises an intermediate pentaerythritol diphosphite having a spiro isomer as shown in the following formula (III),



(III)

a caged isomer shown in the following formula (IV),



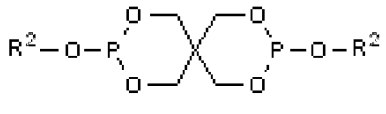
(IV)

unreacted monophosphite and side reaction products wherein R^1 is selected from the group consisting of wherein R^1 is selected from the group consisting straight-chain or branched C_{1-20} alkyl groups, C_{5-7} cycloaliphatic groups and C_{6-11} substituted derivatives thereof, straight-chain or branched C_{2-30}

alkenyl groups, C_{6-18} aryl groups, C_{7-40} alkylaryl groups and C_{7-40} arylalkyl groups and blends thereof;

(b) removing reaction products other than said intermediate pentaerythritol diphosphite from said first reaction mixture; and

(c) transesterifying said intermediate pentaerythritol diphosphite with an alcohol selected from the group consisting of C_{8-22} alkanols, C_{8-22} alkenols, phenols, C_{7-40} alkylaryl alcohols and C_{7-40} arylalkyl alcohols to form a second reaction mixture which comprises a final pentaerythritol diphosphite of formula (VI)



(VI)

where R^2 is selected from the group consisting of C_{8-22} alkyls, C_{8-22} alkenyls, phenyl, C_{7-40} alkylaryls and C_{7-40} arylalkyls.

[c2] 2. The method of claim 1 which further comprises the step of separating said intermediate pentaerythritol diphosphite from said first reaction mixture to produce a purified intermediate pentaerythritol diphosphite.

[c3] 3. The method of claim 2 wherein said monophosphite is selected from the group consisting of trimethyl phosphite, triethyl phosphite and triphenyl phosphite.

[c4] 4. The method of claim 3 wherein said monophosphite is triphenyl phosphite.

[c5] 5. The method of claim 4 wherein said intermediate pentaerythritol diphosphite is diphenyl pentaerythritol diphosphite.

[c6] 6. The method of claim 1 wherein a ratio of said monophosphite to said pentaerythritol is about 2 moles of monophosphite per mole of pentaerythritol.

[c7] 7. The method of claim 1 which further comprises an alkaline catalyst.

[c8] 8. The method of claim 1, wherein said amount of said alkaline catalyst ranges from about 0.01 weight percent to about 5 weight percent, based on the intermediate pentaerythritol diphosphite.

[c9] 9. The method of claim 1 wherein said alcohol is 2,4-dicumyl phenol.

[c10] 10. The method of claim 1, wherein said first transesterification reaction pressure is in a range from about 0.01 mm Hg to about 100 mm Hg.

[c11] 11. The method of claim 10 wherein said first transesterification reaction temperature is in a range from about

70°C to about 105°C.

[c12] 12. The method of claim 11 wherein said first reaction mixture further comprises a solvent.

[c13] 13. The method of claim 12, wherein said solvent is selected from the group consisting of C_6- C_{20} aromatic hydrocarbons and C_6- C_{20} aliphatic hydrocarbons and blends thereof.

[c14] 14. The method of claim 13, wherein said solvent is added in an amount ranging from about 10 weight percent to about 200 weight percent of said intermediate pentaerythritol diphosphite.

[c15] 15. The method of claim 11 wherein said intermediate pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 percent.

[c16] 16. The method of claim 15 wherein said intermediate pentaerythritol diphosphite has a yield of greater than 95 percent, based on the monophosphite.

[c17] 17. The method of claim 2 wherein said separating step comprises distillation of said reaction mixture sufficient to purify said intermediate pentaerythritol diphosphite to a purity of at least 99 percent.

[c18] 18. The method of claim 17 wherein the purity of said

intermediate pentaerythritol diphosphite is at least 99.9 percent.

[c19] 19. The method of claim 2 further comprising the step of separating said second pentaerythritol diphosphite from said second reaction mixture.

[c20] 20. The method of claim 19 wherein said step of separating said second pentaerythritol diphosphite from said second reaction mixture comprises a step of distillation.

[c21] 21. The method of claim 19 wherein said alcohol is selected from the group consisting of 2,4-di-t-butylphenol and 2,4-dicumylphenol.

[c22] 22. The method of claim 21 wherein said alcohol is 2,4-dicumylphenol.

[c23] 23. The method of claim 19 wherein said final pentaerythritol diphosphite is selected from the group consisting of bis(2,4-dicumylphenyl) pentaerythritol diphosphite and bis (2,4-di-t-butylphenyl) pentaerythritol diphosphite.

[c24] 24. The method of claim 19 wherein said second temperature is in a range of about 130°C to about 170°C.

[c25] 25. The method of claim 24 wherein said second pressure is in a range of about 0.01 mm Hg to about 100

mm Hg.

[c26] 26. The method of claim 19, wherein the amount of said alcohol ranges from about 2 moles to about 8 moles per mole of said intermediate pentaerythritol diphosphite.

[c27] 27. The method of claim 19, which further comprises a second alkaline catalyst.

[c28] 28. The method of claim 27, wherein the amount of said second alkaline catalyst is in a range of about 0.01 weight percent to about 5 weight percent, based on the final pentaerythritol diphosphite.

[c29] 29. The method of claim 19, wherein said final pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 mole percent.

[c30] 30. An improved method for synthesizing pentaerythritol diphosphites comprising the steps of:

(a) transesterifying pentaerythritol with a monophosphite to form a first reaction mixture which comprises an intermediate pentaerythritol diphosphite having a spiro and a caged isomer and unreacted monophosphite and side reaction products at a temperature below 125°C;

(b) removing reaction products other than said intermediate pentaerythritol diphosphite from said first

reaction mixture; and

(c) transesterifying said intermediate pentaerythritol diphosphite with an excess of an alcohol to form a second reaction mixture which comprises a final pentaerythritol diphosphite at a temperature below 175°C, said final pentaerythritol diphosphite having a spiro content in excess of 90 mole percent without purification by recrystallization.

[c31] 31. The method of claim 30 which further comprises the step of separating said intermediate pentaerythritol diphosphite from said first reaction mixture to produce a purified intermediate pentaerythritol diphosphite.

[c32] 32. The method of claim 31wherein said monophosphite is selected from the group consisting of trimethyl phosphite, triethyl phosphite and triphenyl phosphite.

[c33] 33. The method of claim 32 wherein said monophosphite is triphenyl phosphite.

[c34] 34. The method of claim 33 wherein said intermediate pentaerythritol diphosphite is diphenyl pentaerythritol diphosphite.

[c35] 35. The method of claim 30 wherein a ratio of said monophosphite to said pentaerythritol is about 2 moles of monophosphite per mole of pentaerythritol.

[c36] 36. The method of claim 30 which further comprises an alkaline catalyst.

[c37] 37. The method of claim 30, wherein said amount of said alkaline catalyst ranges from about 0.01 weight percent to about 5 weight percent, based on the intermediate pentaerythritol diphosphite.

[c38] 38. The method of claim 30 wherein said alcohol is 2,4-dicumyl phenol.

[c39] 39. The method of claim 30, wherein said first transesterification reaction pressure is in a range from about 0.01 mm Hg to about 100 mm Hg.

[c40] 40. The method of claim 39 wherein said first transesterification reaction temperature is in a range from about 70°C to about 105°C.

[c41] 41. The method of claim 40 wherein said first reaction mixture further comprises a solvent.

[c42] 42. The method of claim 41, wherein said solvent is selected from the group consisting of C_6- C_{20} aromatic hydrocarbons and C_6- C_{20} aliphatic hydrocarbons and blends thereof.

[c43] 43. The method of claim 42, wherein said solvent is

added in an amount ranging from about 10 weight percent to about 200 weight percent of said intermediate pentaerythritol diphosphite.

[c44] 44. The method of claim 30 wherein said intermediate pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 percent.

[c45] 45. The method of claim 44 wherein said intermediate pentaerythritol diphosphite has a yield of greater than 95 percent, based on the monophosphite.

[c46] 46. The method of claim 31 wherein said separating step comprises distillation of said reaction mixture sufficient to purify said intermediate pentaerythritol diphosphite to a purity of at least 99 percent.

[c47] 47. The method of claim 46 wherein the purity of said intermediate pentaerythritol diphosphite is at least 99.9 percent.

[c48] 48. The method of claim 31 further comprising the step of separating said second pentaerythritol diphosphite from said second reaction mixture.

[c49] 49. The method of claim 48 wherein said step of separating said second pentaerythritol diphosphite from said second reaction mixture comprises a step of distillation.

[c50] 50. The method of claim 48 wherein said alcohol is selected from the group consisting of 2,4-di-t-butylphenol and 2,4-dicumylphenol.

[c51] 51. The method of claim 50 wherein said alcohol is 2,4-dicumylphenol.

[c52] 52. The method of claim 48 wherein said final pentaerythritol diphosphite is selected from the group consisting of bis(2,4-dicumylphenyl) pentaerythritol diphosphite and bis (2,4-di-t-butylphenyl) pentaerythritol diphosphite.

[c53] 53. The method of claim 48 wherein said second temperature is in a range of about 130°C to about 170°C.

[c54] 54. The method of claim 53 wherein said second pressure is in a range of about 0.01 mm Hg to about 100 mm Hg.

[c55] 55. The method of claim 48, wherein the amount of said alcohol ranges from about 2 moles to about 8 moles per mole of said intermediate pentaerythritol diphosphite.

[c56] 56. The method of claim 48, which further comprises a second alkaline catalyst.

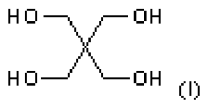
[c57] 57. The method of claim 56, wherein the amount of said

second alkaline catalyst is in a range of about 0.01 weight percent to about 5 weight percent, based on the final pentaerythritol diphosphite.

[c58] 58. The method of claim 48, wherein said final pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 mole percent.

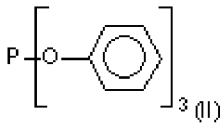
[c59] 59. An improved method for synthesizing bis(2,4-dicumylphenyl) pentaerythritol diphosphite comprising the steps of:

(a) transesterifying pentaerythritol of formula (I)



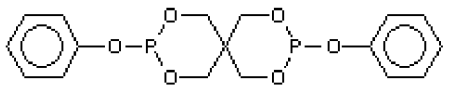
(I)

with triphenyl phosphite of formula (II)



(II)

to form an intermediate reaction mixture comprising diphenyl pentaerythritol diphosphite of formula (V)

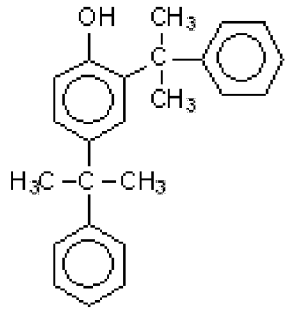


(V);

(b) removing phenol and reaction products other than diphenylpentaerythritol diphosphite from said intermediate reaction mixture;

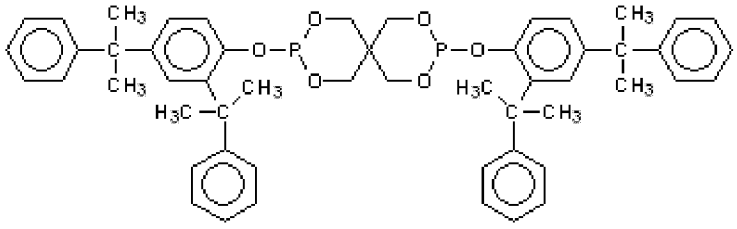
(c) transesterifying said intermediate pentaerythritol

diphosphite with 2,4-dicumyl phenol of formula (VII)



(VII)

to form a second reaction mixture which comprises a final pentaerythritol diphosphite bis(2,4-dicumylphenyl) pentaerythritol diphosphite of formula (VIII)



(VIII); and

(d) removing phenol from said second reaction mixture.

[c60] 60. The method of claim 59 wherein a ratio of said triphenyl phosphite to said pentaerythritol is about 2 moles of triphenyl phosphite per mole of pentaerythritol.

[c61] 61. The method of claim 59 which further comprises an alkaline catalyst for said first transesterification reaction.

[c62] 62. The method of claim 61, wherein said amount of said alkaline catalyst ranges from about 0.01 weight percent to about 5 weight percent, based on the intermediate

diphenyl pentaerythritol diphosphite.

[c63] 63. The method of claim 62, wherein said first transesterification reaction pressure is in a range from about 0.01 mm Hg to about 100 mm Hg.

[c64] 64. The method of claim 63 wherein said first transesterification reaction temperature is in a range from about 70°C to about 105°C.

[c65] 65. The method of claim 64 wherein said first reaction mixture further comprises a solvent.

[c66] 66. The method of claim 65, wherein said solvent is selected from the group consisting of C_6- C_{20} aromatic hydrocarbons and C_6- C_{20} aliphatic hydrocarbons and blends thereof.

[c67] 67. The method of claim 66, wherein said solvent is added in an amount ranging from about 10 weight percent to about 200 weight percent of said intermediate diphenyl pentaerythritol diphosphite.

[c68] 68. The method of claim 67 wherein said intermediate diphenyl pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 percent.

[c69] 69. The method of claim 68 wherein said intermediate diphenyl pentaerythritol diphosphite has a yield of

greater than 95 percent, based on the triphenyl phosphite.

[c70] 70. The method of claim 59 wherein said step of removing phenol from said first reaction mixture comprises distillation.

[c71] 71. The method of claim 70 wherein said step of removing phenol from said reaction mixture purifies said intermediate diphenyl pentaerythritol diphosphite to a purity of at least 99 percent.

[c72] 72. The method of claim 71 wherein the purity of said intermediate diphenyl pentaerythritol diphosphite is at least 99.9 percent.

[c73] 73. The method of claim 70 wherein said step of removing phenol from said second reaction mixture comprises distillation.

[c74] 74. The method of claim 73 wherein said second temperature is in a range of about 130°C to about 170°C.

[c75] 75. The method of claim 74 wherein said second pressure is in a range of about 0.01 mm Hg to about 100 mm Hg.

[c76] 76. The method of claim 75, wherein the amount of said 2,4-dicumyl phenol ranges from about 2 moles to about

8 moles per mole of said intermediate diphenyl pentaerythritol diphosphite.

[c77] 77. The method of claim 76, which further comprises a second alkaline catalyst for said second transesterification step.

[c78] 78. The method of claim 77, wherein the amount of said second alkaline catalyst is in a range of about 0.01 weight percent to about 5 weight percent, based on the bis(2,4-dicumylphenyl) pentaerythritol diphosphite.

[c79] 79. The method of claim 78, wherein said final bis(2,4-dicumylphenyl) pentaerythritol diphosphite comprises a spiro isomer content of greater than 90 mole percent.

[c80] 80. The method of claim 1, which further comprises a step of recycling at least a portion of said reaction products other than said intermediate pentaerythritol diphosphite from said first reaction mixture.

[c81] 81. The method of claim 80, wherein said step of recycling comprises a step of recycling phenol.

[c82] 82. The method of claim 80, which further comprises a step of recycling at least a portion of said reaction products other than said final pentaerythritol diphosphite

from said second reaction mixture.

[c83] 83. The method of claim 82, wherein said step of recycling comprises a step of recycling 2,4-dicumyl phenol.

[c84] 84. The method of claim 30, which further comprises a step of recycling at least a portion of said unreacted monophosphite or said side reaction products.

[c85] 85. The method of claim 84, wherein said step of recycling comprises a step of recycling phenol.

[c86] 86. The method of claim 84, which further comprises a step of recycling at least a portion of said alcohol.

[c87] 87. The method of claim 86, wherein said alcohol is 2,4-dicumyl phenol.

[c88] 88. The method of claim 59, which further comprises a step of recycling at least a portion of said first reaction mixture selected from the group consisting of triphenyl phosphite, phenol and reaction products.

[c89] 89. The method of claim 88 which further comprises a step of recycling at least a portion of said second reaction mixture selected from the group consisting of 2,4-dicumyl phenol and phenol.